

STANDARD OPERATING PROCEDURE

Parr Acid Digestion Vessel	Version: 1.0	Date: 04/14/11	Written by: Whitney Schmidt
Page 1 of 8			Reviewed by: Geneva Laurita-Plankis Authorised by: M.A. Subramanian

Subramanian Research Group, Department of Chemistry, Oregon State University

Chemistry Department Safety Office: Gilbert Hall Room 153
Subramanian Research Group: 7-6750
Emergency Medical Services: 911
Campus Student Health Center: 7-9355
Poison Control: 9-1-800-222-1222
OSU Environmental and Health Safety: 7-2273
Campus Security: 7-7000

DISCLAIMER:

This Standard Operating Procedure (SOP) was developed based on instrument manuals, manufacturer specifications, and laboratory experience to provide guidance to Subramanian Lab users in performing the activities defined in this document, in a consistent and standardized manner. This document does not contain regulatory or statutory requirements unless specified.

The authors of this document have made every attempt to present the information in a clear and concise manner for all users of the Subramanian Lab. However, the Subramanian Lab is not responsible for the misuse or misinterpretation of the information presented in this SOP. Under no circumstances shall the Subramanian Lab be liable for any actions taken or omissions made by users of this SOP.

In general, this document should NOT be used in place of a manufacture's instrument manual, warnings and instructions pertaining to use and safety of the specified equipment. This specific document may be used as a supplement to procedure and quick reference to safety but does not replace the information found with the manufactures documentation for the specified equipment. The Subramanian Lab reserves its right to change or suspend any or all parts of this document.

USE OF AUTOCLAVES

1. Introduction

This SOP discusses the procedure and safety guidelines for the use of Teflon-lined, stainless steel laboratory autoclaves manufactured by Parr Instrument Company (formally named acid digestion vessel).

The autoclave is a closed stainless steel vessel with an internal cup and lid made of Teflon. The instrument can be charged with reagents, and closed. Under external heating, the contents will be raised to higher temperatures and pressures than within an unsealed container. The autoclaves are specifically designed for harsh chemicals, high temperatures and high pressures.

2. Scope

This procedure applies to all staff, students and visitors of the Subramanian Research Group in the Department of Chemistry at Oregon State University that work in the laboratory and have the potential to use the autoclave for solvothermal or hydrothermal experiments.

STANDARD OPERATING PROCEDURE

Parr Acid Digestion Vessel	Version: 1.0	Date: 04/14/11	Written by: Whitney Schmidt
Page 2 of 8			Reviewed by: Geneva Laurita-Plankis Authorised by: M.A. Subramanian

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3. Safety

- Personnel who operate the autoclave must be trained to understand proper packaging, loading, labelling, as well as operation and emergency procedures.

General precautions:

- Personal protective equipment (PPE) including safety glasses or face shield, protective laboratory gloves, closed-toed footwear, and laboratory coats must be worn when charging or opening autoclaves; and heat-insulating gloves must be put on when removing the autoclaves from the oven.
- Only trained laboratory personnel can operate the instrument.
- **Reagents used in the vessel must not react to release gas.** This will lead to excessive pressure build-up.
- Before every use both the stainless steel shell and the Teflon liner should be visually inspected for cracks, pitting, rust, metal creep or excessive wear. **Steel shells that are cracked or flawed in any way must be discarded.** Worn or distorted Teflon liners should be replaced.
- When charging the autoclave with reagents, the Teflon liner should never be filled more than 66 percent (filling fraction, ~15 mL). This will ensure that there is sufficient headspace for vapour formation and fluid expansion when the vessel is heated.
- Nitric acid should not be used with organic materials to prevent the formation of nitro explosives.
- Reactions which are highly exothermic or which release large quantities of gas (such as an oxidant and an organic compound) should not be performed in the autoclave.
- **Perchloric acid should NEVER be used in the vessel.**
- Do not overheat the autoclave. The maximum temperature is 250 °C.
- Do not exceed the pressure limit for each run. It should be verified if the pressure expected is within the limits of the device. Check relevant vapour pressure tables (Parr Instrument Company Acid Digestion Vessels Operating Instruction Manual, pg 8).
- This model has a spring-assisted pressure plate system that will release energy should the pressure within the vessel exceed the autoclaves specifications. Nothing must ever be done to inhibit the function of the pressure plate system. The spring must be replaced if it shows signs of excessive wear.
- After heating, the autoclaves must be allowed to naturally air cool on a heat-resistant plate.
- **The autoclaves should NEVER be quenched in water.**
- After use, the autoclaves must not be opened until they are fully cool. Even then, they should be opened with care because the contents may still be under pressure.

Risk of implosion or explosion:

Materials should not be autoclaved if they:

- Contain radioactive material.

STANDARD OPERATING PROCEDURE

Parr Acid Digestion Vessel Page 3 of 8	Version: 1.0	Date: 04/14/11	Written by: Whitney Schmidt Reviewed by: Geneva Laurita-Plankis Authorised by: M.A. Subramanian
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Subramanian Research Group, Department of Chemistry, Oregon State University

- Contain or are suspected of containing hazardous chemicals (solvents or corrosives).
- Chemicals that will become unstable/volatile at elevated temperatures.
- Contaminated sharps.

Incident Response:

- If any injury occurs seek first aid or, if necessary, seek medical assistance.
- If clothing is soaked in hot water/steam, remove clothing and cool the injured part in cool water.
- Place a notice on the autoclave indicating that it is not to be used until the cause of the incident is determined, procedures enacted to prevent future incidents, and the autoclave is deemed safe for operation.

Spill Clean Up:

- Visibly inspect the autoclave and inside of the oven prior to cleaning any ruptured equipment.
- Spills may occur from a boil-over or breakage of containers.
- No operation of the autoclave is allowed until the spill is cleaned up.
- The operator is responsible for clean-up of spills. Contain the spilled material using materials from the spill kit to absorb or contain the spill.
- Wait until the autoclave and materials have cooled to room temperature before starting the cleanup inside the autoclave.
- Review the Material Safety Data Sheets (MSDS), if appropriate, to determine the protective equipment, spill cleanup, and disposal protocols that are necessary.
- Clean the equipment and work area in order to collect and remove all spilled materials. Dispose of the waste following the protocol appropriate for the material. If materials have been intermingled, follow the cleanup and disposal protocol for the most hazardous component of the mixture.

5. **Training and Competency**

The trainee must have already mastered an understanding of and have been given the instruction in the use of autoclaves by an approved trainer (the instrument supervisor or any trained member of the Subramanian Research Group). Competency will be assessed by close observation of the trainee by the instrument supervisor or an approved trainer. The training records are attached at the end of this SOP, pg7.

7. **Equipment and Maintenance / Handling and Storage / Labelling**

- Autoclaves are marked with unique numbers.

STANDARD OPERATING PROCEDURE

Parr Acid Digestion Vessel Page 4 of 8	Version: 1.0	Date: 04/14/11	Written by: Whitney Schmidt Reviewed by: Geneva Laurita-Plankis Authorised by: M.A. Subramanian
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Subramanian Research Group, Department of Chemistry, Oregon State University

- **Only matched autoclave parts are to be used. No mix-matching of lids to bodies allowed.**
- No person shall operate the autoclave unless the autoclave is in good repair.
- Users are not to make repairs. Autoclaves shall be maintained and repaired by qualified persons.
- If the autoclave does not operate exactly as expected, a notice shall be placed on the autoclave indicating that it is not to be used until the problem is diagnosed and corrected.
- Autoclaves are to undergo visual inspection prior to use, and stringently inspected every six (6) months.

Signage

- Signage indicating “Hazardous Process Underway” must be displayed when autoclaves in use.
- Each oven has signage indicating the temperature, DO NOT change the temperature without verifying that the oven is not in use. Replace the signage to reflect the set temperature.

8. Operating Procedures

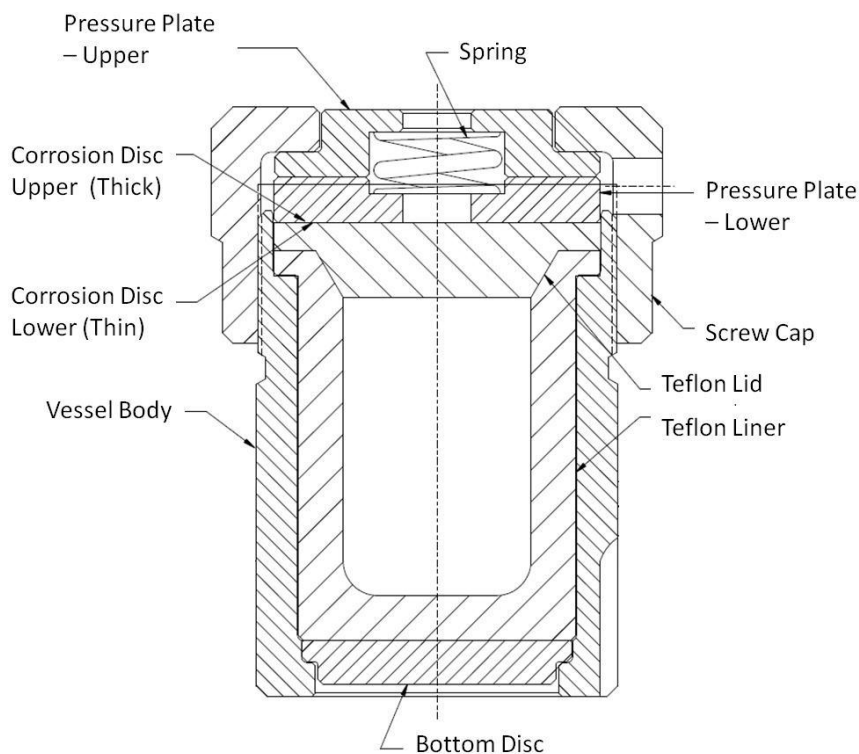


Figure 1. Parr acid digestion vessel model 4749.

STANDARD OPERATING PROCEDURE

Parr Acid Digestion Vessel Page 5 of 8	Version: 1.0	Date: 04/14/11	Written by: Whitney Schmidt Reviewed by: Geneva Laurita-Plankis Authorised by: M.A. Subramanian
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Subramanian Research Group, Department of Chemistry, Oregon State University



Figure 2. Holding fixture and Hook spanner.

Loading Autoclaves:

1. Place the desired reagents into the *Teflon Liner* (charging).
 - Do not mix incompatible materials
 - **Do not overload; leave sufficient room for steam circulation. The maximum volume allowed is 66 percent and 1 g total of solid material.**
2. Place the stainless steel *Bottom Plate* into stainless steel *Autoclave Body* and ensure that the *Bottom Plate* is in the correct place.
3. Ensure that the lip of the *Teflon Liner* is free from any liquid/solid by gently cleaning with a Kimwipe.
4. Place the *Teflon Lid* onto the *Teflon Liner*. Nothing should impede good contact between the lid and liner.
5. Gently place the charged *Teflon Liner* into the *Autoclave Body*.
6. Place the *Corrosion Discs* (*first thin then thick*) onto the *Teflon Lid*
7. Place the *Lower Pressure Plate* onto the corrosion plates with the spring cavity facing upwards the place the *Spring* into the spring cavity of the *Lower Pressure Plate*. Only undamaged springs should be used.
8. Place the *Upper Pressure Plate* onto the *Spring/Lower Pressure Plate/Autoclave Body* assembly spring cavity side down.
9. Screw the stainless steel *Screw Cap* onto the *Autoclave Body* assembly until finger tight.
10. KEEP VESSEL ON BENCHTOP TO TIGHTEN, do not pick up to tighten. Spilling can occur out of the cup.
11. Then secure holding fixture into a vice and tighten the lid with a spanner.
 - Do not over-tighten – no more than 1/8th turn after tight.

STANDARD OPERATING PROCEDURE

Parr Acid Digestion Vessel Page 6 of 8	Version: 1.0	Date: 04/14/11	Written by: Whitney Schmidt Reviewed by: Geneva Laurita-Plankis Authorised by: M.A. Subramanian
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Subramanian Research Group, Department of Chemistry, Oregon State University

- Do not cross-thread the *Autoclave Body* and *Screw Cap*.
12. Transport the autoclave assembly/s to the reaction ovens (Room 209).
 - Do not excessively tilt the autoclaves. Spilling into assembly may occur.
 - Warning – charged autoclaves are heavy.
 13. Commence hydrothermal/solvothermal reaction by placing the autoclave assembly/s into the reaction oven. The oven may be preheated or cold. Wear heat-insulating gloves when placing autoclaves into a preheated oven.
 14. **Record materials used in the reaction and vessel number in the Log, found in the Hydrothermal Vessel Instructions and Log book.**

Unloading Autoclaves:

1. Put on appropriate PPE including heat-insulating gloves and safety glasses or face shield prior to handling heated autoclaves.
2. Open oven door and gently remove the autoclave/s and place them on a thermal-resistant plate or ceramic brick to cool down.
 - **Allow the autoclave/s to cool down naturally.**
 - **DO NOT QUENCH THE TEMPERATURE BY PLACING THE VESSELS INTO COLD WATER.**
 - Shut down the oven if necessary prior to removal of autoclave/s.
3. After completely cool, secure the holding fixture in a vice and unscrew the *Screw Cap* of autoclave using the spanner.
 - Only open an autoclave when completely cooled.
4. Gently remove the *Pressure Plate/Spring/Teflon Liner* assembly from the *Autoclave Body* by slowly pressing the *Bottom Plate* upwards. Remove the *Pressure Plates*, *Corrosion Discs* and *Spring* prior to removing the *Teflon Liner* and *Teflon Lid*.
5. Remove the reactant from the *Teflon Liner* and process as required.
6. When complete, clean the Teflon Liner and dry. Place the autoclave assembly into the designated storage box. Ensure that all matched autoclave parts are stored together (screw cap, pressure plates, autoclave body, etc.).

9. **Controls and Calibrations**

Stringent visual inspections of the autoclaves are to be performed every six (6) months to examine their structural integrity. The autoclave should be taken out of use if there is any evidence of damage, deterioration or metal creep on the thread of the autoclave body, lid or other stainless steel components.

10. **Waste Disposal**

Solvent Disposal: Dispose of solvents in the appropriate waste container (e.g. Water soluble waste solvent, water insoluble waste solvent, halogenated waste solvent).

STANDARD OPERATING PROCEDURE

Parr Acid Digestion Vessel Page 7 of 8	Version: 1.0	Date: 04/14/11	Written by: Whitney Schmidt Reviewed by: Geneva Laurita-Plankis Authorised by: M.A. Subramanian
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Subramanian Research Group, Department of Chemistry, Oregon State University

11. Relevant Documents / References

- Advanced Porous Materials Laboratory, School of Chemistry, The University of Melbourne, SOP Version 1.0, 07/14/10.
- *Parr Operating Instructions: Parr Acid Digestion Bombs*, Parr Instrument Company, No. 249M.

12. Signage / Summaries / Templates

Competency Training Records Form – Attached, see page 7.

- Copies of this form are to be stored and filed and in the Subramanian Research Group Hydrothermal Vessel Instruction and Log book.

